

Standard Operating Procedure for the Sampling of Particulate-Phase and Dissolved-Phase Organic Carbon in Great Lakes Waters

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Standard Operating Procedure for the Sampling of Particulate-Phase and Dissolved-Phase Organic Carbon in Great Lakes Waters

1.0 SCOPE AND APPLICATION

- 1.1 This Standard Operating Procedure describes the sampling of Great Lakes Waters for particulate-phase organic carbon (POC) and dissolved-phase organic carbon (DOC).
- 1.2 Samples of lake water are collected and passed through a 0.7- μ m pore-size glass fiber filter. POC is operationally defined as the mass of organic carbon retained on the filter per unit volume of water, and DOC is the material that passes through the filter.

2.0 SAFETY AND WASTE HANDLING

- 2.1 Refer to GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended) and individual instrument procedural operations manuals for specific details on applicable 1) personal health and safety issues; 2) instrumental, chemical, and waste handling procedures and; 3) accident prevention. This applies to all EPA personnel, EPA contractors or federal, state, or local government agencies, and persons who operate or are passengers onboard US EPA GLNPO vessels during all activities and surveys.
- 2.2 All applicable safety and waste handling rules are to be followed. These include proper labeling and disposal of chemical wastes. Over-board discharges of chemical wastes are forbidden.
- 2.3 During sampling, caution, common sense, and good judgement should dictate appropriate safety gear to be worn in any given situation on deck. Hard hats, gloves, and steel-toed shoes must be worn in working conditions where there is a possibility of injury to the head, hands, or feet; however, if in doubt, please ask the Chemical Hygiene Officer.
- 2.4 Collecting samples in cold weather, especially around cold water bodies, carries the risk of hypothermia and frostbite. Sampling team members should wear adequate clothing for protection in cold weather. For specific information regarding sampling during cold conditions, please refer to the US EPA GLNPO *Standard Operating Procedures for Winter Operations* (December 1994, or as amended).
- 2.5 Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Sampling team members should carry an adequate supply of water or other liquids for protection against dehydration in hot weather.
- 2.6 Work vests must be worn while working on the fantail and Rosette deck.

3.0 SUMMARY OF PROCEDURE

- 3.1 Great Lakes water samples are collected at pre-determined sampling stations and depths *via* either a submersible pump or Rosette sampler.
- 3.2 The water is then filtered under vacuum through ashed 47-mm diameter glass fiber filters in an all-glass filtration apparatus. The samples are acidified during the filtration to remove inorganic carbonates.
- 3.3 The POC is retained on the filter and frozen at -10°C until analysis. The filtrate is collected and promptly analyzed for DOC in a ship-board laboratory.

4.0 DESCRIPTION OF APPARATUS

- 4.1 Water samples (typically 1-4 liters for open-lake locations) are collected from an over-board pump or Rosette sampler. Ashed glass fiber filters are supported in a commercially-available, all-glass, 350-mL vacuum filtration apparatus.
- 4.2 Two filtration apparatuses are attached, side-by-side, to ring stands. Samples are filtered simultaneously in duplicate. Tygon tubing (3/8" ID) is used to connect the filtration flasks to an oil-less vacuum pump. The equipment needed are listed in Table 1.

5.0 PREPARATION OF FILTERS AND REAGENTS

5.1 Preparation of Filters

- 5.1.1 Filter preparation should take place as close to the start of the survey as possible.
- 5.1.2 Filters are to be handled only with stainless steel forceps. Filters that are mishandled after the ashing procedure (5.1.4) should be discarded.
- 5.1.3 47-mm diameter GF/F filters (0.7- μ m pore-size) are placed individually in aluminum foil envelopes, dull side of foil facing inward, with three sides folded closed. The fourth side is left open to allow gases to escape from the envelope during ashing.
- 5.1.4 The filters are stacked in a muffle furnace and ashed for four hours at 450°C.
- 5.1.5 Upon removal from the muffle furnace, the envelopes are sealed on the fourth side.
- 5.1.6 Fifty envelopes containing individual filters are placed into a Ziploc bag and the bag is labeled with the date and initials of the analyst who prepared the filters.

5.2 Preparation of Reagents

- 5.2.1 A solution of 0.2N HCl is prepared by transferring 17 mL of concentrated HCl (16.1 N) to a 1000-mL volumetric flask and diluting to the mark with organic-free, distilled, reagent water. Transfer the solution to a 1-L Teflon squeeze bottle.

6.0 FILTRATION PROCEDURE

- 6.1 Using stainless steel forceps, place one 47-mm GF/F filter onto the fitted glass support of the sampling apparatus. Place the glass funnel on top of the filter and secure with the clamp. Label the Great Lake name, station number, sampling depth, and date onto the aluminum foil envelope.
- 6.2 Collect the lake water sub-samples from the submersible pump hose or Rosette sampler. Allow the overboard pump line to flush for 15-30 minutes. Collect the lake water into a 4-L Cubitainer or four 1-L Teflon bottles. Rinse the container(s) twice with approximately 1 liter of lake water before collecting the sample. If the lake water is to be collected from the Rosette, rinse the container(s) with only 200-300 mL of lake water to ensure there is enough remaining to establish a significant particulate load on the filter (See section 6.7).
- 6.3 Measure the volume of lake water to be filtered in a graduated cylinder, or mark four 1-L Teflon bottles at the 1-liter level. Prior to filling, rinse the bottles, or cylinder, twice with approximately 100 mL of lake water.

- 6.4 Connect the vacuum pump to the filtration flask. Pour a measured volume of lake water into the glass filtration funnel. Turn on the vacuum pump. Maintain the vacuum between 5-10 inches of Hg during filtration.
- 6.5 After approximately 300 mL of lake water has been filtered, turn off the vacuum pump. Rinse the 200 mL DOC glass sample bottle several times with filtrate and collect approximately 150 mL of the filtrate. Label the Great Lake name, station number, sampling depth and date onto the DOC bottle. Collect the filtrate before step 6.6.
- NOTE:** Step 6.6 must be done before all the lake water is filtered to ensure that the distribution of the particles on the filter is not disturbed.*
- 6.6 Turn on the vacuum pump, and continue pouring lake water into the funnel until sufficient material has been collected (see section 6.7). Just before the last portion of the lake water has been filtered, squirt approximately 5 mL of 0.2 N HCl solution into the funnel.
- 6.7 The volume of lake water required to produce a reliable POC measurement (i.e., an amount of material that is within the analytical instrument's linear range) will vary with lake station location, depth, and time of year. For open-lake, oligotrophic conditions, typically 2-4 liters will provide enough material. For near-shore locations, or meso-eutrophic and eutrophic conditions, lake water volumes in the range of 200-500 mL are typical. A filter that becomes visibly loaded with particles and a flow of water through the filter that drops significantly are evidence that sufficient particulate material has been collected.
- 6.8 After the lake water has been filtered, rinse the sides of the funnel with approximately 20 mL of reagent water and filter this rinse. Turn off the vacuum pump.
- 6.9 Remove the funnel. Using stainless steel forceps, fold the filter in half and place back it into the labeled aluminum foil envelope. Place groups of foil envelopes in a labeled Ziploc bag and store at -10°C. Record the Great Lake name, station number, sampling depth, volume filtered, analyst, date, and time of day on the POC/DOC Sampling Log Sheet.
- 6.10 Empty the remaining filtrate from the filtration flask.
- 6.11 Rinse the filtration funnel, fitted glass support, filtration flask, and the container(s) with reagent water.
- 6.12 Re-assemble the filtration apparatus.
- 6.13 Place aluminum foil covers over the filtration funnels.

7.0 QUALITY CONTROL

- 7.1 A duplicate sample will be filtered in parallel at least once during the sampling of each Great Lake.
- 7.2 A POC/DOC matrix blank will be collected, in duplicate, at the beginning of each survey of the Great Lakes, and at least once during the sampling of each Great Lake. A TSS matrix blank is collected by filtering 1 liter of reagent water. A DOC matrix blank consists of the filtrate from a POC matrix blank. The matrix blanks are processed identically to Great Lakes water samples.
- 7.3 A POC field blank will be collected, in duplicate, at the beginning of each survey of the Great Lakes, and at least once during the sampling of each Great Lake. A POC field blank is prepared by taking a filter out of the foil envelope, placing it onto the fritted glass support of a clean filtration apparatus, wetting the filter with reagent water and assembling the filtration apparatus. The apparatus is disassembled, and the filter is removed and processed in the same manner as a sample. There is no field blank for DOC.

- 7.4 Two trip blanks for POC will be processed after the survey has ended. This is done by placing two filters in their unopened foil envelopes into the Ziploc bag and processing these filters like samples. There is no DOC trip blank.
- 7.5 DOC samples are analyzed promptly, in a ship-board laboratory, during the course of a survey.
- 7.6 Because POC/DOC are parameters which are ancillary to the determination of hydrophobic organic contaminants (HOCs), the POC/DOC samples during an organics survey are taken simultaneous to the HOC samples. Therefore when a HOC matrix blank, field blank or duplicate sample is collected, a POC/DOC matrix blank, field blank or duplicate sample will also be collected.

TABLE 1: LIST OF FILTRATION EQUIPMENT

Quantity	Equipment	Source or Equivalent
2	Oil-less vacuum pump	Schuco 5711-130
6	Teflon wash bottle	Cole-Parmer N-06052-60
2	350-mL, all-glass Filtration apparatus	Nucleopore
2	Stainless Steel Forceps	NA
2	Support/ring stand for filtration apparatus	NA

Miscellaneous (some quantities depend on number of samples)

- 47-mm GF/F filters (0.7- μ m pore-size) Whatman 1825-47
- Cubitainers
- Tygon tubing (3/8"ID)
- 200-mL glass bottles for DOC
- permanent markers
- Ziploc freezer bags
- Aluminum foil